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Jane Dee Hull, Governor
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DATE: March 21, 1996

TO: Laboratory Director and QA Manager

FROM: Wesley B. Press, Acting Bureau Chief

SUBJECT: Information Update #25

NOTE: If any problems occur with this web site, please call 1-800-952-0374 or (602) 255-3454 extension 205, 221 or 222. Thank You.

1. The last information update #24, contained the following question and answer. We felt that the answer needed further clarification.

   QUESTION: What are the detection levels required by the State for phase II & V SOC compounds?

   ANSWER: For an individual point of entry sample the lab must be able to see 50% of the MCL (except for four of the compounds). These four compounds are atrazine, dibromochloropropane, ethylene dibromide, and di(2-ethylhexyl)pthalate. For these four compounds, the Lab must be able to quantitate down to the MCL for each compound.

   For composite samples the state regulations refer back to the federal regulations. The federal regulations require that the detection limit of the method used for analyses be less than one-fifth of the MCL.

   Further clarification:

   For a single point of entry sample, the trigger level for increased monitoring by a water system is any concentration greater than or equal to 50% of the MCL except for the four compounds listed above which have trigger levels at any concentration greater than the MCL. Therefore the laboratories must be able to quantitate down to these trigger levels.

   Compositing of a maximum of five samples are allowed and it must be done in a licensed laboratory. For compositing samples for regulated SOCs, the following 2 criteria must be met;

   a. The detection limit of the method used for the analysis is less than one-fifth of the MCL.

b. If a composite sample is analyzed for SOCs, the lab must be able to quantitate down to the MDL for the composite sample {refer to 40 CFR, Part 141.24, paragraph (h)(18) and the Arizona Department of Environmental Quality Drinking Water Rules, R18-4-219, Appendix B, for the required detection limits}. This is due to the fact that for composite samples the trigger level for increased monitoring is any concentration greater than or equal to the MDL.

2. Our Office received a Memorandum from William R. Diamond, Director, Drinking Water Standards Division, Office of Ground Water Drinking Water, USEPA, concerning the approval of the Quanti-Tray Test for total coliforms and E. Coli under the Total Coliform Rule and the Surface Water Treatment Rule. A copy of the memo is attached.

3. "Extraction of Appendix IX BNAs, Pesticides & PCBs by Accelerated One-Step TM Liquid-Liquid Extractor/Concentrator with Analysis by GC/MS & GC\ECD" is an acceptable alternate technique to EPA Method 3520, Continuous Liquid-Liquid Extraction. As per the letter from Barry Lesnik, Chemist, Methods Section, RCRA Organic Methods Program Manager, to James E. Carl, Project Supervisor, Product Development, Corning Inc, this modified extraction apparatus reduces the solvent volume and shortens extraction time without adversely affecting the performance of Method 3520. The extraction method can be obtained from Dave Black at Corning Inc., 1-(800) 222-7740.

4. HACH Method 10014 DPD is approved for determining ultra low level Residual chlorine in waste waters for NPDES permit.

5. Following are some of the additional questions discussed at the Round Table Discussion on Inorganic and Microbiology methods (2/9/96).

QUESTION: Total / Fecal Coliform by Membrane Filtration:
Blanks are required by the method, but what corrective action should be taken if the blank has a few colonies and there are no colonies in the samples?

ANSWER:

a. If the colonies on the blank membrane filter are non-coliform, investigate to avoid in the future, and report the no growth water sample as negative for coliform.
b. If the colonies on the blank membrane filter are coliform and/or fecal coliform, take corrective action by investigating cause and alert data user by stating findings on final report. Suggest resampling.

QUESTION: How common is it to have typical colonies (metallic green sheen) that do not confirm?

ANSWER: From a discussion with Don Reese, Section Manager, Environmental Microbiology, State Laboratory Services, Arizona Department of Health Services, typical metallic sheen colonies are rare in drinking water but are more common in ambient water (streams, lakes and bathing beaches).

QUESTION: Can transfer sticks be sterilized in the autoclave? We don't have an oven.

ANSWER: No, transfer sticks cannot be sterilized in the autoclave. Buy sterile sticks or use metal loops which must be flamed prior to and after culture transfers. If sterilization is a problem, sterile disposable inoculating loops can be ordered from various vendors. Our office does not endorse any particular product, but the following are examples of these products. VWR Scientific: Difco disposable inoculating loop, catalog# DF1906-95 and DF1906-96; disposable inoculating loop, (loop/needle combination), catalog# 50806-300 or 50806-344. HACH Company: disposable inoculating loop, catalog# 22454-25.

QUESTION: If a Colilert sample is negative but turbid must it be reported as turbid and be resampled?

ANSWER: No, as per Dale Ohnmeiss, Environmental Program Supervisor, Arizona Department of Environmental Quality.

QUESTION: Why can't we pipet samples "line-to-line" for microbiological testing?

ANSWER: If, for example, 0.1 mL samples need to be pipetted from a 1 mL pipet which is properly marked in 0.1 mL increments, line to line pipetting is permitted.

QUESTION: HPC : Can a result be reported as < 1CFU/ml? If so, when?

ANSWER: Yes, HPC results can be reported as < 1CFU/mL, when:

a. no colonies are found on either of the duplicate sample plates, or
b. 1 CFU is found on only one of the plates and 1 mL of the sample was plated.

QUESTION: What are the laboratory requirements for reporting micro results to ADEQ?

ANSWER: The laboratories are no longer required by ADEQ to report the Maximum Contaminant Level (MCL) violations in the drinking water samples within 72 hours. The owners of the utilities are primarily responsible for reporting the MCL violations to ADEQ within 48 hours after the receipt of the analytical reports. One exception being nitrate, which must be reported within 24 hours. The laboratories are ultimately responsible for the test results and they may continue to report all the test results to ADEQ, if the client so desires.

QUESTION: Does each vessel in the Colilert test have to be checked for color using the color comparator after 24 hr. of incubation? It is quite obvious when a yellow color is produced by coliforms.

ANSWER: If the yellow color is obvious, the comparator check is not necessary. All the presumed negative samples must be checked with the comparator to confirm the negative result. The color comparator must be used after 24 hr. of sample incubation to confirm the absence of slight color variation. Important: the color comparator solution must be stored in a clear plastic container. If a slight color variation is detected, but the comparison with the comparator is not definitive, then the sample must be incubated for an additional 4 hrs at 350 C.

QUESTION: We don't have a 600 C incubator and are using our 350 C incubator to incubate spore strips/ampules used in validating our autoclave efficiency. Is this OK? It gives good

results.

ANSWER: No, this is not acceptable. Spore strips or suspensions must be incubated at the manufacturer's recommended temperature, usually 600 C.

QUESTION: What is the difference between Dissolved, Suspended, Total and Total Recoverable Metals?

ANSWER:
Dissolved Metals: Those elements which will pass through a 0.45 um membrane filter.
Suspended Metals: Those elements which are retained by a 0.45 um membrane filter.
Total Metals: The concentration determined on an unfiltered sample following vigorous digestion or, the sum of the dissolved plus suspended concentrations.
Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

The current Federal regulations still classify metals contamination using three categories: total, dissolved and suspended metals.

The following information was provided to us by Ted Martin, Research Chemist, USEPA/NERL, Cincinnati, Ohio, and it describes the reasons why a change in sample preparation and definitions has occurred in drinking water and has been proposed for wastewater.

CHANGE IN EPA SAMPLE PREPARATION FOR METALS DETERMINATIONS - PROPOSED FOR NPDES

In the original "total metal" digestion (paragraph 4.1.3 on page METALS-6) given in Methods for Chemical Analysis of Water and Wastes, nitric acid was added to the sample and refluxed until the digestion was complete, indicated by a light color digestate. The determined analyte concentration following this digestion was reported as "total metal". This term was used because the sample was not filtered prior to digestion and the determined concentration reflected the combined metal concentration of the sample - the "dissolved metal" concentration + the "suspended metal" concentration. However, to report the concentration as "total metal" was in some cases a misnomer because the digestate was not clear, indicating that the "total sample" was not completely solubilized and available for analysis.

In Methods for the Determination of Metals in Environmental Samples - Supplement I, the digestion of unfiltered aqueous samples has been altered. In the revised procedure (Method 200.2) a specific amount of acid (HNO3 + HCl) is added to the sample and refluxed for 30 minutes following evaporation. The determined analyte concentration following digestion is now defined as "total recoverable". EPA has adopted this term because it is a more appropriate definition of the analyte concentration available for analysis following acid solubilization. This revised procedure is a single uniform digestion which can be used prior to analysis by direct aspiration flame atomic absorption, and is included in the EPA methodology for the stabilized temperature graphite furnace (EPA Method 200.9 revision 2.2), for ICP-AES (EPA Method 200.7 revision 4.4), and for ICP-MS (EPA Method 200.8 revision 5.3).

Using NBS 1645 and other reference samples EPA has found the revised digestion procedure in Supplement I comparable to the previously accepted "total metals" procedure. EPA believes

the revised procedure is sufficiently vigorous to render analytes available for NPDES compliance monitoring requiring a total metal measurement (dissolved + suspended) where the sample is not filtered and digested prior to analysis.

QUESTION: What procedure do I use to digest samples for metals in wastewater for NPDES permits? How do I report the final results?

ANSWER: Digest the samples using the methods referenced in the 40CFR, part 136.3, Table IB: For samples analyzed by ICP, use one of the digestion procedures found in the Method 200.7 published in the 40 CFR, Part 136, appendix C; For the samples analyzed by other procedures listed in the above table, use one of the digestion procedures found in the metals section of "Methods for Chemical Analysis of Water and Wastes, 1979 and 1983."

The final concentrations determined by any of the above procedures are reported as "total."

QUESTION: What are the appropriate digestion procedures for methods 200.7, 200.8, 200.9 in drinking water? Is it okay to report the result as a "total" metal?

ANSWER: Use the digestion procedures that are included with the above methods. The results may be reported as "total" metal.

QUESTION: Can there be a combined digestion procedure for the SW-846 methods, i.e. 6010A and 7000 series?

ANSWER: The EPA is currently looking into establishing a single digestion procedure that could be used for the SW-846 methods.

QUESTION: Can SM 3113B be used for thallium analysis on wastewater? Can 200.9 be used? If not what will happen when 279.2 is withdrawn?

ANSWER: Method 279.2 is scheduled to be withdrawn as of July 1, 1996 for drinking water analysis. As it stands now, 200.9 will be the only method approved for drinking water as of July 1, 1996.

You will still be able to use 279.2 for thallium analysis on wastewater samples for NPDES permits. SM 3113B is not approved for thallium analysis. 200.9 cannot be used for waste water, it is only proposed.

QUESTION: Method 200.7 does not specify a calibration range. Can methods 200.7 and 6010A be combined in one instrument run using the same calibration?

ANSWER: Yes. When running either method the analyst must determine the linear range for each analyte on the instrument. The sample results cannot be reported if they exceed 90% of top end of the linear range. Be aware that the major sample constituents (ex. Ca) may exceed the linear range and will need to be diluted.

QUESTION: My multitask ICP run (sequential) for 200.7 has poor Ag recovery, possible cause & solution?

ANSWER: You need sufficiently high concentrations of chloride in order to stabilize the Ag. Because of this, method 200.7 in 40 CFR, Part 136, recommends that samples for the determination of silver be digested. Digestion procedure given in section 9.4 in 200.7 is the preferred sample preparation technique. Be careful during the digestion, not to reduce the volume below 15 mls as this may cause the silver to precipitate out. Samples digested by this procedure should be able to hold up to 2 mg/L of silver. Also, during the analysis be sure to use a rinse acid that has the same acid concentration as the samples and standards.

6. Our first Round Table Discussion was quite successful in addressing questions and concerns in the Inorganic and Microbiology areas. We are scheduling our second Round Table Discussion on Organic GC Volatile methods. A couple of our surveyors from the Environmental Laboratory Licensure Section and staff from the State Laboratory will be present to answer/clarify questions that you may have about any of the methods and related questions. This will be held **Friday, April 2, 1996 from 1:30 - 3:30 pm at 3443 North Central, 9th floor conference room.**

   The areas to be covered are the approved methods, quality control, trouble shooting, maintenance of instrumentation and any other relevant questions. We request that you fax your questions to us prior to this date, in case we need to call the EPA for further clarification. Please fax your questions to Prabha Acharya at 255-1070. You are welcome to bring your questions with you if you cannot fax them ahead of time. Our training room can hold up to 35 people so please RSVP with Cristy Finan at 255-3454 to ensure availability of space.

   Please note that we will not be validating any parking. Paid parking is available adjacent to the building on the street level.

7. If you have any questions regarding the Updates, or if you have any technical questions that need clarification, please call or send e-mail to Prabha Acharya, Program Manager, Technical Resources and Training at the Laboratory Licensure. A table of contents to all the Information Updates published is also available.